Methods for Mitigating Growth of Laser-Initiated Surface Damage on DKDP Optics at 351nm

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Methods for mitigating growth of laser-initiated surface damage on DKDP optics at 351nm

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ABSTRACT

We report an experimental investigation of mitigating surface damage growth at 351nm for machine-finished DKDP optics. The objective was to determine which methods could be applied to pre-initiated or retrieved-from-service optics, in order to stop further damage growth for large aperture DKDP optics used in high-peak-power laser applications. The test results, and the evaluation thereof, are presented for several mitigation methods applied to DKDP surface damage. The mitigation methods tested were CW-CO_2 laser processing, aqueous wet-etching, short-pulse laser ablation, and micro-machining. We found that micro-machining, using a single crystal diamond tool to completely remove the damage pit, produces the most consistent results to halt the growth of surface damage on DKDP. We obtained the successful mitigation of laser-initiated surface damage sites as large as 0.14mm diameter, for up to1000 shots at 351nm and fluences in the range of 2 to 13J/cm^2, ~11ns pulse length. Data obtained to-date indicates that micro-machining is the preferred method to process large-aperture optics.

Keywords: laser damage, DKDP, surface damage growth mitigation, CO_2 laser processing, micro-machining

1. INTRODUCTION

Surface damage initiated on machine finished DKDP surfaces during high-peak-power irradiation at 351nm encompasses only a fraction of the clear aperture area. However, studies have shown that the damage can grow with the number of shots at 351nm at laser fluences above ~5J/cm^2, 11ns pulse length [1,2]. A substantial increase in the useful lifetime of the optics can be achieved by stopping damage growth, thus mitigating obscuration caused by growing damage. Ideally this can be accomplished by eliminating the damage sites while they are small and returning the surface to its undamaged state. We explored several methods to mitigate the growth of UV-laser-induced damage on DKDP crystals. Other studies at this Laboratory have focused on elucidating the mechanisms for initiation [3] and growth [4] of surface damage on DKDP optics, whereas this effort seeks to identify and validate a leading method to successfully terminate the growth of such damage on DKDP optics.

Prior to testing for successful mitigation methods, it was necessary to determine how surface damage on DKDP actually grows at 351nm. Since there was little data available at 351nm prior to this work, we performed experiments to establish the

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growth behavior of surface damage on DKDP. These experiments were done for both mechanically produced damage such as indents and scratches, and laser-produced damage. While these experiments did not provide sufficient data to establish the conditions for predictable surface damage growth, we determined that surface damage may grow in three distinct ways. Rapid, potentially catastrophic growth is rare, but it can be triggered when the fluence exceeds $8J/cm^2$, at both 3ns and 11ns pulse durations. Surface damage more commonly grows at fluences greater than $6J/cm^2$, by a process of 'fragmentation' within the existing site, usually including the ejection of particles. In some cases, the 'fragmentation' occurs only during a fluence change, then stops altogether when the fluence is stable. In other cases, growth by 'fragmentation' may be triggered after many shots at a fixed fluence. These unpredictable growth responses are shown graphically in Fig. 1 for six almost identical mechanical indent sites.

The following sections describe the experiments and results of damage growth tests for the four mitigation methods considered in this study; CW-CO_2 laser processing, aqueous wet-etching, short-pulse laser ablation, and micro-machining. The objective was to determine which methods could be applied to pre-initiated or retrieved-from-service optics, in order to stop further damage growth for large-aperture DKDP optics used in high-peak-power laser applications. A specific goal was to obtain sufficient data and information to enable the selection of a single approach for processing large-scale optics. The results show that micro-machining, using a small, high speed motor and a single-crystal diamond bit that completely removes the damage pit, consistently halts the growth of surface damage on DKDP. We have used this method to process surface damage sites on a small-scale DKDP optic to demonstrate feasibility for processing large-scale optics.

2. EXPERIMENTAL

In our experimental program, we tested the growth of mitigated mechanical and laser-initiated damage pits, as well as the intrinsic growth behavior of the mitigation pits themselves. It was necessary to determine that the pits formed by the mitigation methods on undamaged surfaces did not cause other damage or grow with repeated illumination. In fact, this was the case for some of methods that were tested.

The survey experiments used the same type of DKDP sample; diamond tool machine-finished DKDP, 50mm x 50mm x 10mm thick. The damage pits were arranged on the exit surface of these samples in an array of 9 spots spaced by ~10mm. Mechanically produced indents or scratches of various sizes and depths were produced by a Nano-Instruments, Model XP nano-indenter. For the laser-initiated pits, to avoid inducing bulk damage within the DKDP, we used a tight focused beam to place an array of 9 nominally uniform, equally spaced damage pits on the exit surface of DKDP samples. Each laser-initiated damage site was produced by a single pulse from a frequency-tripled Nd:YAG laser at 355nm, with a 0.9mm beam diameter, at an average fluence of ~$75J/cm^2$ and a pulse length ~7.5ns. Each of the mitigation methods had distinctive surface pits, with different shapes and sizes, as shown in Fig.2. Later experiments to qualify the micro-machining mitigation method, were done using 150mm x 150mm x 10mm thick samples, which had naturally occurring, laser-initiated surface damage sites on them. The experiments were done at 351nm, at both ~3ns and ~11ns pulse lengths.

The growth tests of the survey samples were all carried out in a vacuum chamber operating in the range from ~10^{-5}torr to 5torr, in the slab laser facility at LLNL [1]. The sites were tested for growth using the frequency-tripled Nd-glass laser output, producing a 4mm x 6mm flat-top beam at 351nm, with ~11ns pulse width at 1-12 J/cm^2. The fluence is ramped to condition the crystal against bulk damage, in all cases. The damage sites are illuminated by the laser at a rate of 0.5Hz and images produced by the transmitted laser light are recorded after each laser shot. Any visible change of the test site during laser illumination indicated an ineffective mitigation method. The qualification tests were done at 2.5 – 10torr of nitrogen gas. Some tests were done in the slab laser facility using the same laser as the survey tests and others were done in the Optical Science Lab (OSL) facility, using a frequency tripled Nd-glass laser, which produced a 3cm diameter flat-top beam at 351nm, with ~3ns pulse width at 1-8J/cm^2.

3. CW-CO₂ LASER EXPERIMENTS AND RESULTS

CO_2 lasers have been used successfully to mitigate surface damage growth on fused silica at 351nm [5] but we were not aware of any attempts to use CO_2 laser heating to treat existing damage on DKDP. We explored using a CO_2 laser for mitigating surface damage growth on DKDP. Experiments were done using a Parallax, Inc. 20W CW-CO₂ laser and an external reed-switch to give variable pulse duration. This laser was operated at less than 1W CW with a focused Gaussian beam, a $1/e^2$ beam diameter of ~0.1mm, for 1-100ms pulse duration. The laser beam locally melted and evaporated the DKDP, typically producing shallow, uneven pits with raised edges (see Fig.2a). The average pit depth (~4–15μm) depended on both laser power and the defocus length.

The initial growth tests for the CO_2 laser-produced pits were performed in the slab laser facility at 351nm, ~11ns. The pits on both bare surface and indent damages changed only while the fluence was being ramped, but essentially no change was observed for hundreds of subsequent shots at a fixed fluence. However, in later tests carried out in OSL at ~3ns pulse duration, the CO_2 laser-produced pits continued 'fragmentation' damage at fixed fluence shots, after ramping. The typical mode of pit damage during the fluence ramp was by 'fragmentation' within the pit, including particle ejection.

We conclude that the CO_2 laser treatment is not a viable mitigation method because 'fragmentation' and particle ejection were not inhibited. However, this approach is worthy of further investigation if the source of absorption at 351nm by the modified DKDP is determined and eliminated.

4. WATER-ETCHING EXPERIMENTS AND RESULTS

We tested wet-etching as a potential mitigation method on DKDP. Since liquid water quickly dissolves DKDP crystals, it is possible to remove an existing damage on the crystal surface by applying a small droplet of water to the damage spot and then removing the solution before it dries. However, this is complicated because water droplets tend to spread due to surface tension, resulting in a much larger affected area than the original damage size. We have employed means to produce and deliver very small droplets to control the spread of liquids at the crystal surface and to remove dissolved material. One method was to deliver a small droplet of water through a capillary surrounded by a co-axial, evacuated tube. Two variations were used; one with and one without oil, which was employed to limit the spread of water on the crystal surface and to inhibit drying. Another method was to deliver a jet of moist gas to the crystal surface through a micro-jet. The moisture in the gas jet condenses on the crystal and etches it. This process can also be configured with a co-axial vacuum tube to enable solution removal. These methods are depicted in Figs. 3a,b,c and the pits that they produce on the crystal surface are shown in Figs. 2b,c,d.

The water etch pits were tested for stability at 351nm, ~11ns, in the slab laser facility. All such pits on bare surfaces and on indents damaged by 'fragmentation' at fluences >6J/cm². They continued to 'fragment' with subsequent laser shots, but the damage did not propagate beyond the edges and it was generally confined to the area within the pit itself (see Fig. 4). The edges of the etched pits contain some re-crystallized DKDP and meta-phosphates, which may have enhanced absorption and contribute to the damage.

From these preliminary test results, we conclude that wet-etching is not a viable mitigation method for DKDP because 'fragmentation' and particle ejection were not inhibited. This method would be the most difficult to apply for treatment of large-scale optics because it involves handling liquids near the optics, necessitating special considerations to prevent permanent damage of the optic.

5. SHORT-PULSE LASER ABLATION EXPERIMENTS AND RESULTS

In principle, surface damage pits on DKDP can be removed by ablating the affected material using an ultra-short pulse laser. We attempted to use femtosecond laser pulses for this purpose as a possible mitigation method. We employed a Ti-sapphire laser that produced 150fs pulses at 416nm wavelength, focussed at the crystal surface. A single pulse caused ablation of the DKDP, forming a smooth, shallow pit with very little debris or collateral damage (see Fig. 2e). Such pits were generally stable when illuminated by 351nm, ~11ns laser pulses at fluences up to 12J/cm^2. However, when the femtosecond laser pulses are applied to existing damage pits on DKDP, they grow rapidly instead of ablating. Although we chose to abandon this method based on the preliminary results, we believe that more work is likely to produce laser conditions that will remove damage pits on DKDP without growth. This method would be preferred due to the ease of applying the laser beam to treat multiple sites on a large optic.

6. MICRO-MACHINING EXPERIMENTS AND RESULTS

An option for mitigating surface damage growth is the complete removal of the damage pit by micro-machining. We used a small, high-speed (65,000RPM) turbine motor and tiny bits to make shallow cuts into the crystal surface. Pits on DKDP that are made using carbide and diamond bits are shown in Fig. 2f and 2g, respectively. Such pits were tested for stability in the same manner as the pits made by the other methods. We found that the pits or digs made on the DKDP surface were very stable when illuminated by laser pulses at 351nm. Unlike the other methods, the micro-machined pits survived as many as 1000 laser shots at 351nm, ~11ns, at fluences as high as 12J/cm^2, without damaging or major 'fragmenting'; a few (2 out of 28) sites showed minor 'fragmentation' damage after 600 shots. We also used the single-crystal diamond micro-machining to remove existing surface damage on DKDP, then tested the sites at 351nm, ~11ns for 550 shots at 12J/cm^2, without damage. Additional tests were done in the OSL facility to qualify the single-crystal diamond micro-machining mitigation method at 351nm, 3ns pulse length. No new damage was observed on 10 tested sites for 10 shots at 8J/cm^2. An example of the successful mitigation of an existing surface damage on a DKDP sample is shown in Fig. 5.

7. SUMMARY

A summary of the test results for each of the methods is given in Table 1. None of the tested pits showed rapid, catastrophic growth behavior, although rapid growth of entrance surface damage was observed on some of the same samples. When unmitigated indents damaged, it was by 'fragmentation'. Most mitigated sites damaged by 'fragmentation' within or at the edge of the mitigation pit. The threshold for 'fragmentation' of pits is ~5J/cm^2 and most changes occur during the conditioning ramp between 5-12J/cm^2. Only minor changes occur within the pits at constant fluence. Machined pits are generally non-damaging but some machined with oil damaged by 'fragmentation', usually at a single location on the edge of the pit. Micro-machining is the only method that produces completely stable pits at all of the test conditions.

Successful mitigation of several natural, laser-initiated surface damages was demonstrated on 150mm x 150mm x 10mm samples of machine-finished DKDP samples, at 12J/cm^2, ~11ns, as well as, at 8J/cm^2, ~3ns.

Table 1. Tabulated results of stability tests for mitigation methods applied to bare surfaces and to indent damages on DKDP surfaces. The conditions in these tests were the following: 351nm wavelength, ~11ns pulse length; a conditioning ramp from 1 - 12J/cm^2 in ~50 shots at each site; an additional 550 shots at 12J/cm^2 at each site; an additional 200 shots at ~14J/cm^2 on some sites.

Mitigation method	Observed response of the pit
CO_2 laser on bare surface	'fragmentation' only during fluence ramp
CO_2 laser on mechanical indent	'fragmentation only during fluence ramp
Vac. assisted micro-fount. on bare surface	'fragmentation' mostly at edges of pit
Vac. assisted micro-fount. on mechan. indent	'fragmentation' mostly at edges of pit
Oil assisted micro-fount. on bare surface	'fragmentation' mostly at edges of pit
Oil assisted micro-fount. on mechan. indent	'fragmentation' at indent and at edges of pit
Femto-sec laser on bare surface	'fragmentation' mainly during fluence ramp
Femto-sec laser on mechanical indent	'fragmentation' mainly during fluence ramp
Carbide drill bit on bare surface	no change
Carbide drill bit on mechanical indent	'fragmentation' only where indent was not completely removed
Single-crystal diamond bit on bare surface	no changes
Single-crystal diamond bit on mechan. indents	no changes
Single-crystal diamond bit on laser damage	no changes

8. CONCLUSIONS

Mitigation of surface damage growth on DKDP at 351nm is necessary to prevent 'fragmentation' and particle generation even if rapid, catastrophic growth rarely occurs. Several of the mitigation methods that were tested exhibited some stability for high fluences at 351nm, however, only the micro-machined pits on DKDP are completely stable.

Site-by-site processing of laser damage on DKDP with micro-machining, using a high-speed drill with a single-crystal diamond bit mounted on a micro-positioning stage (see Fig.6), is a feasible mitigation method to apply to large-scale DKDP optics. Based on the results of this study, this is the primary mitigation method recommended for DKDP optics used in high-peak-power applications at 351nm.

ACKNOWLEDGEMENTS

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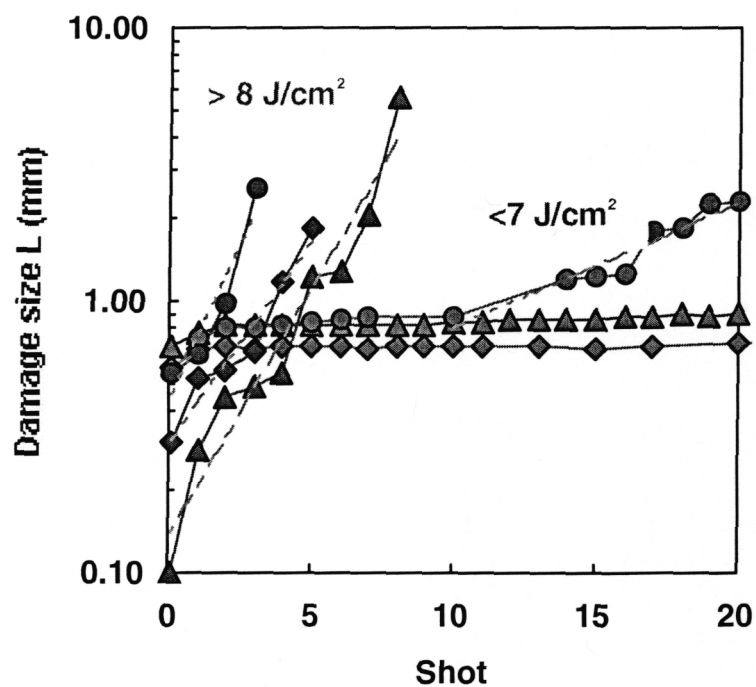
Figure 1. The growth rate of mechanical indent damage on DKDP at 351nm, ~11ns, is not predictable. The graph shows growth responses for 6 nearly identical indent pits on DKDP. Rapid growth is rare but it is sometimes triggered at fluences >8J/cm^2. More commonly, 'fragmentation' occurs during the first few shots at fluences >7J/cm^2, then there is essentially no change for many following shots at constant fluence. Occasionally, slow 'fragmentation' growth may restart after a number of shots.
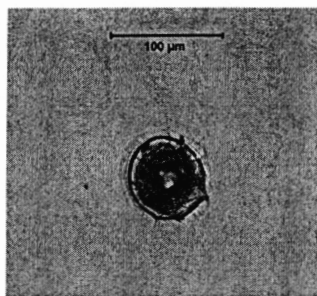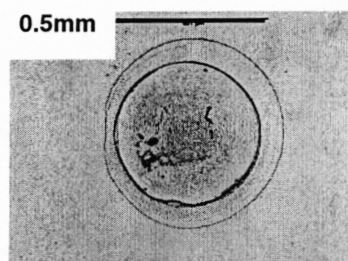
a) After 5 millisecond, 0.23W CO_2 laser pulse
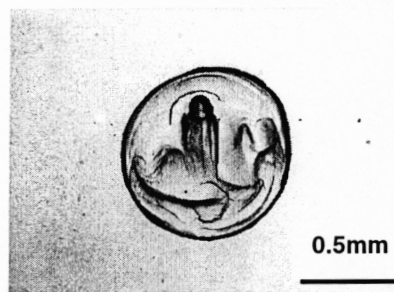
b) After dissolution by water and evaporation
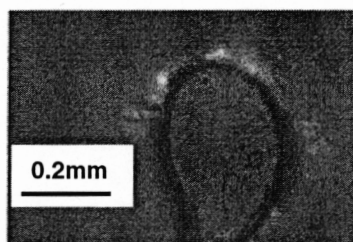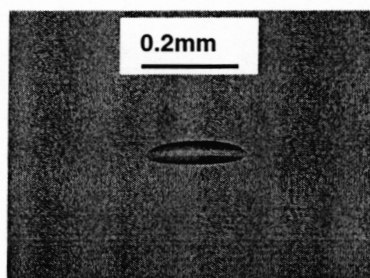
e) After a single 150fs laser pulse

c) After dissolution by water/oil and evaporation
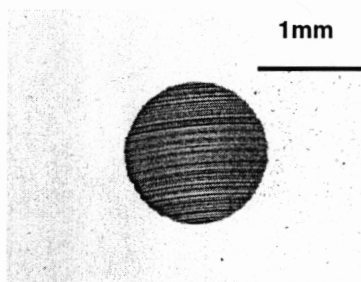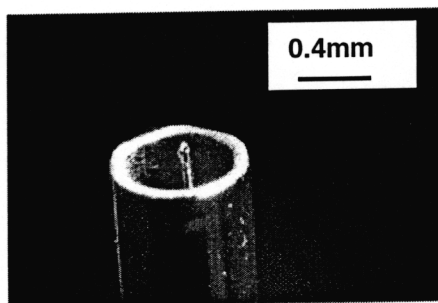
f) After micro-machining with a carbide bit
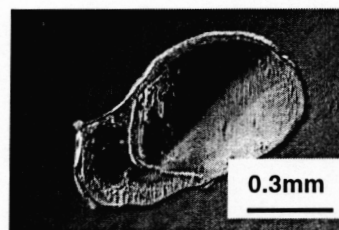
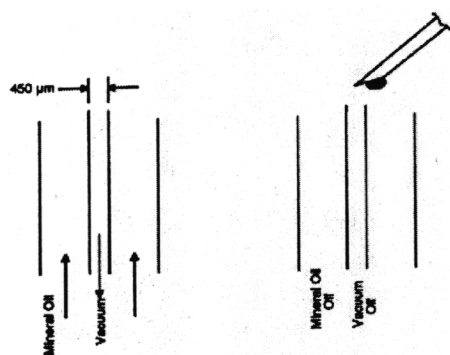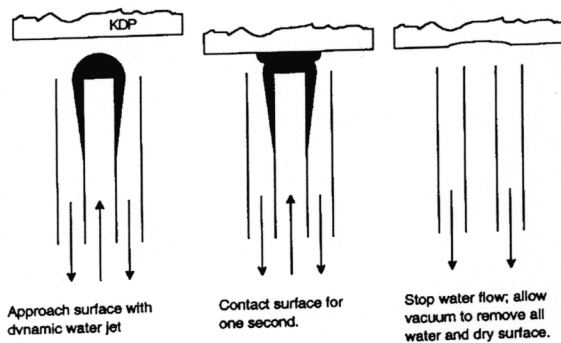d) After dissolution by water from vapor jet

g) After micro-machining with a single-crystal diamond bit

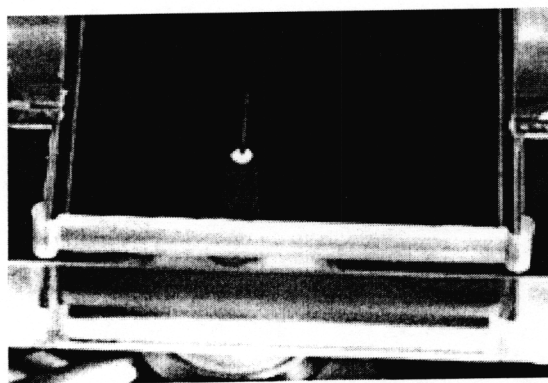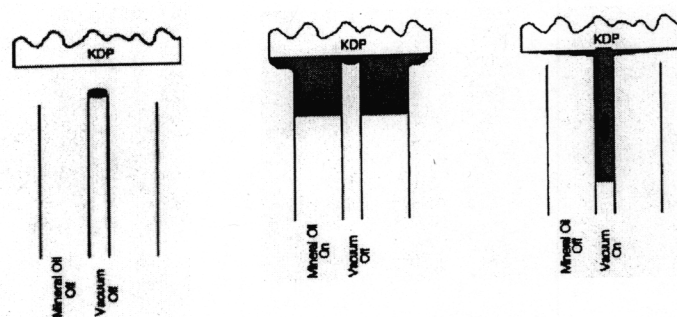Figure 2. Images of pits produced on DKDP surfaces by the mitigation methods that were tested in this study.

0.4mm

KDP

Approach surface with dynamic water jet

Contact surface for one second.

Stop water flow; allow vacuum to remove all water and dry surface.

a) Vacuum-assisted micro-fountain

450 μm

Mineral Oil On

Vacuum On

Mineral Oil Off

Vacuum On

KDP

Mineral Oil Off

Vacuum On

KDP

Mineral Oil On

Vacuum Off

KDP

Mineral Oil Off

Vacuum On

b) Oil-assisted micro-fountain

c) Water vapor jet

Figure 3. Three processes that were used to apply small amounts of water to the DKDP surface for wet-etching; a) vacuum-assisted micro-fountain, b) oil-assisted micro-fountain, and c) water vapor jet.
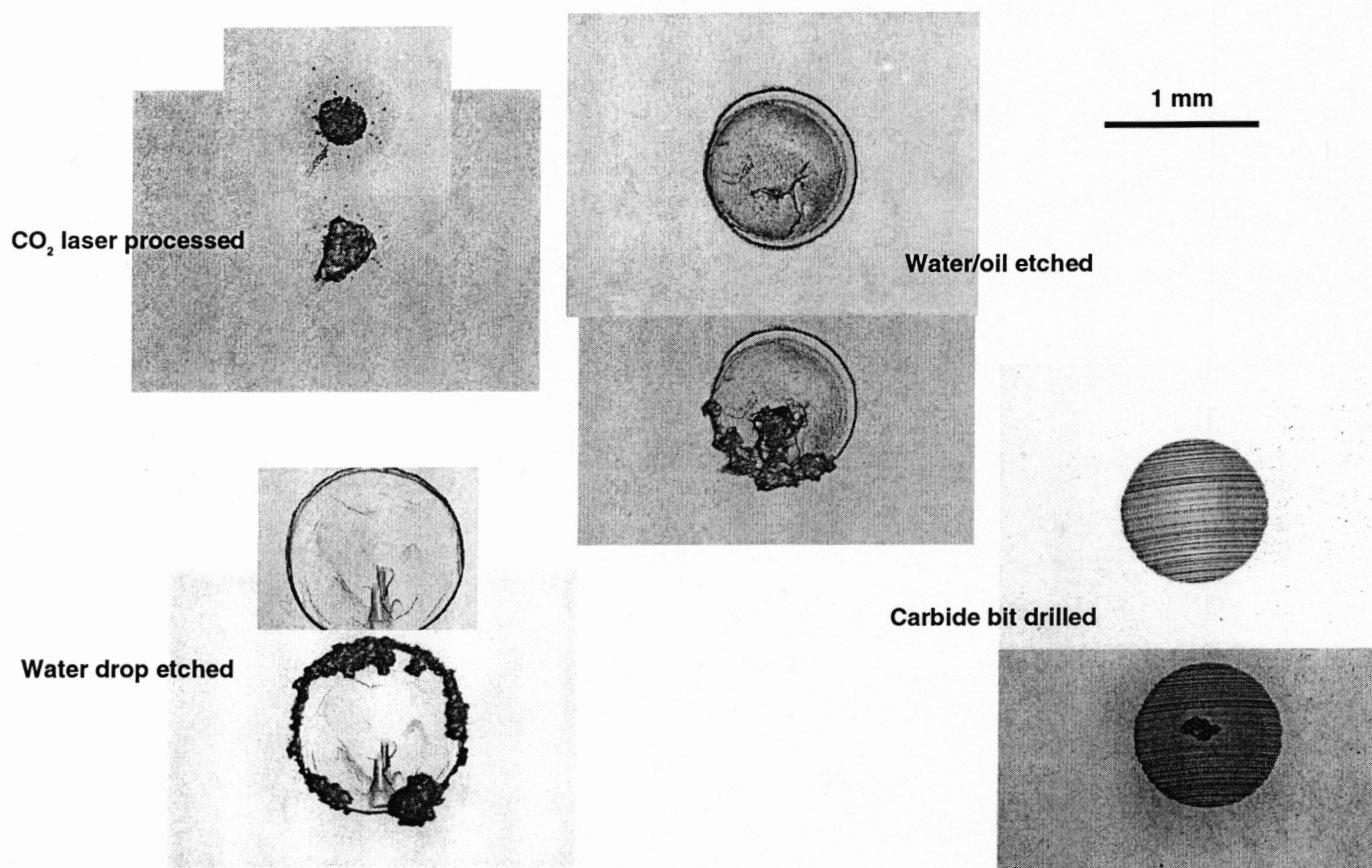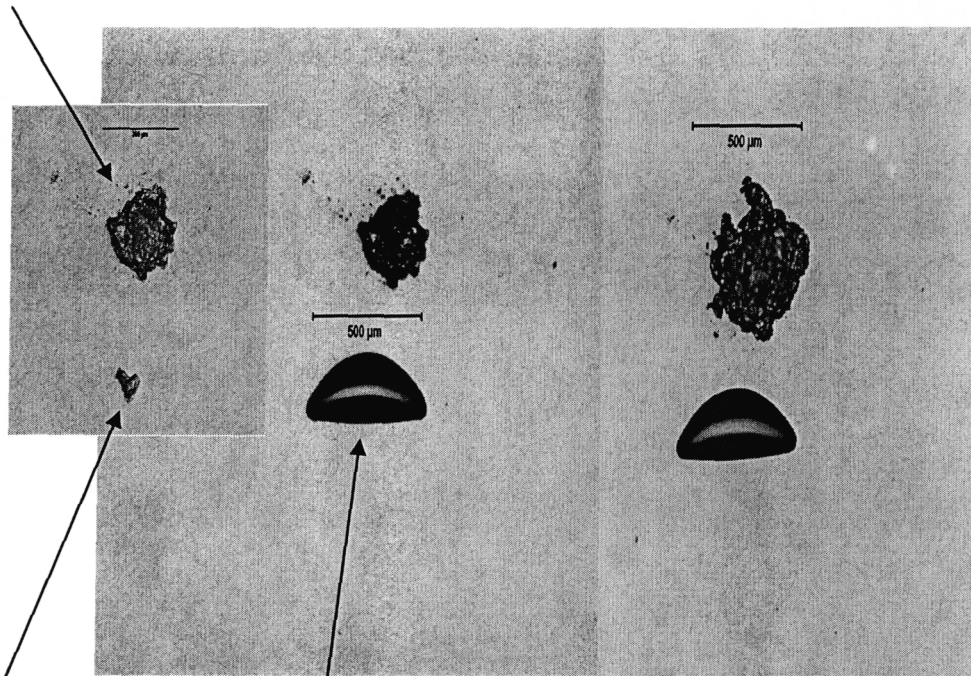
CO₂ laser processed

Water/oil etched

Water drop etched

Carbide bit drilled

1 mm

Figure 4. Before vs. after examples of 'fragmentation' damage that typically occurs during testing of mitigation pits at 351nm, ~11ns, >6J/cm^2.

Damage grown *from a mechanical indent after*
600 shotsof 351nm @12J/cm², 11ns.

Surface damage that was initiated during
testing of the above site, is removed by
micro-machining with a
diamond t

After 600 shots of 351nm @12J/cm², 11ns, there is no
change of the machined pit, while the untouched damage
continues to grow

Figure 5. An example of the successful mitigation of a growing, laser-initiated surface damage on DKDP.
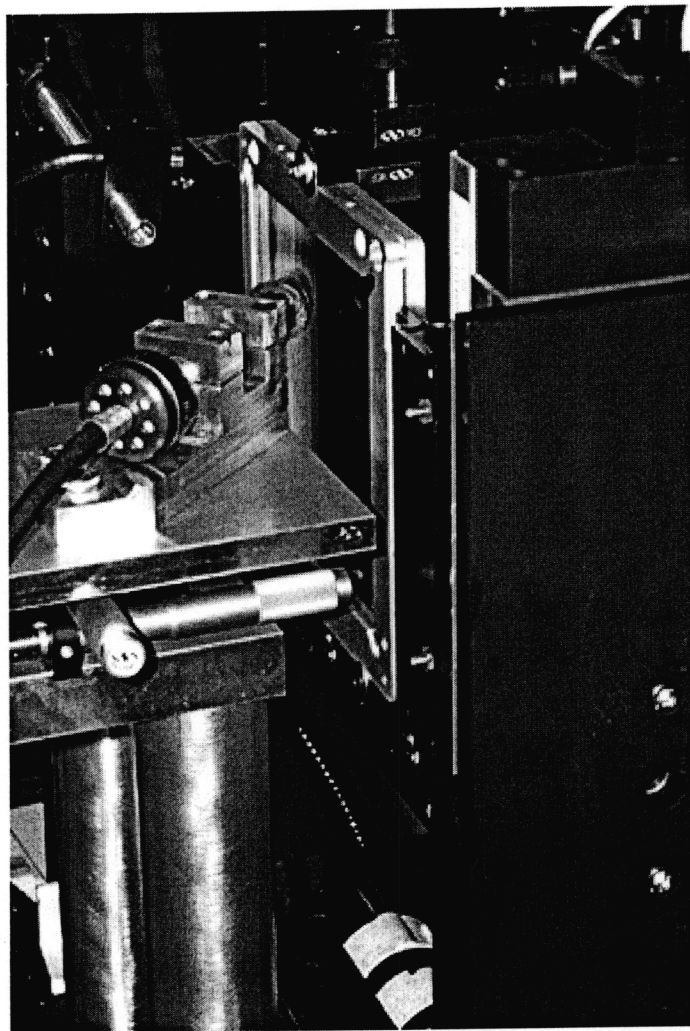
Figure 6. A photo showing a set-up for spot-to-spot processing of surface damages on small-scale DKDP crystals. The mitigation is accomplished by precision positioning of the optic on an x-y stage, and precision positioning of the tool in the z-direction to control the depth of cut.